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MAINTENANCE MANUAL: INSTRUCTIONS FOR SENSOR CLEANING AND FIELD CALIBRATION. THE POTOMAC RIVER WATER POLLUTION MONITORING SYSTEM

A. F. Mentink

Federal Water Pollution Control Administration

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The Potomac River Water Pollution Monitoring System

A.F.Mentink

U.S. DEPARTMENT OF THE INTERIOR
FEDERAL WATER POLLUTION CONTROL ADMINISTRATION

DIVISION OF POLLUTION SURVEILLANCE
Cincinnati, Ohio

September 1966

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SENSOR CLEANING AND

FIELD CALIBRATION

These instructions are a composite of suggestions from the manufacturer and of techniques developed in this laboratory and are intended for the instrumentation system installed on the Potomac River. This instrumentation was designed and developed by the Schneider Instrument Company (formerly Engineering Specialties), 3115 Camargo Road, Madeira, Ohio, and was procured under HEW Contract No. PH27-65-109 and HEW Contract No. PH27-66-33.

PREFACE

Instructions for first-level maintenance (sensor cleaning and field calibration) are covered in this manual. In addition to following these instructions, it is expected that the field technicians retain a log on each system for each monitor. By noting the gradual shift in calibration over several service calls, the technician can readily anticipate sensor replacement. Other levels of maintenance occasionally require electronic engineering background, so it is expected that the Instrumentation Activities, 1014 Broadway, Cincinnati, Ohio, be contacted when conditions related to the second (functional troubleshooting) and third (system expansion) levels arise.

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A. Introduction

One of the most important but yet underemphasized aspects of automatic water quality data acquisition systems is the maintenance of the field instrumentation. As contaminants build up on electrodes, the sensitivity decreases proportionally dependent upon the parameter measured. The more disciplined and rigorous the maintenance program, the greater the probability of obtaining valid and useful data.

B. Levels of Maintenance

The maintenance of a water quality data acquisition system is comprised of three levels: namely, the first level (sensor orientated); the second level (functionally orientated); the third level (system orientated). This section discusses only the first level of maintenance and, as noted below, is subdivided into four basic steps:

1. Initial observation of system.

2. Determination of the electrical calibration check points.

3. (a) Thorough cleaning of the sensors and subsequent measurement of standards (excepting dissolved oxygen);

   (b) Comparison of dissolved oxygen with a Winkler.

4. Final adjustments in both the operate and calibrate mode.

C. Periodicity

The river has unique characteristics at each sampling point, so that the frequency of station visits for sensor cleaning must be determined by field personnel. Initially, however, the format outlined herein should be performed on Tuesday and Friday of each week until it has been demonstrated that a single weekly service call at each station is sufficient. It is evident that a large network of stations will either require an adjustment to this initial plan or additional manpower.

D. Communications with Central Station

During maintenance, invalid data may be telemetered to the central data receiving station. The invalid data must be identified at the central station so that it may be deleted before processing. Since the maintenance at each station should be pursued until the work is completed, which may extend beyond normal working hours, it may be difficult to communicate verbally with data analysts at the central station. Hence, it is suggested that the transmitter test signal be set to 0100 at the start of any maintenance of a given station. This will alert the data analysts to those files which may contain non-representative data; i.e., data generated by measurement of standards,

distilled water, or sensors exposed to air. Since some of the data will be valid, data processing personnel probably will want to filter out all data with 0100 test signal with an editing subroutine before pursuing the analysis. Before this feature in the transmitter is employed, it would be well to discuss its application with those who conduct the data operations.

It is most important to return the transmitter test signal to 1000 millivolts when maintenance has been completed.

E. Parametric Systems

Although the theory associated with these measurements will be described in a separate document, the following instructions can be followed without investigating the theory of each measurement. Patience and care is required to effectively provide a validly calibrated and clean parametric system.

In checking the calibration of each parametric system, all plug-in components should be seated properly in their respective sockets, and the analyzers should be securely fastened in their respective positions to provide proper contact at the blue-ribbon connector.

All sensor leads should be checked and the terminal connections cleaned and fastened securely.

The drawers containing the analyzers should be closed to retain the heat generated by the 40 Watt light bulb thermostatically controlled to provide an internal temperature of 105°F. The 40 Watt lamp is a maintenance accessory as it provides light when either drawer is open, regardless of temperature.

1. pH Parametric System - Introduction

The pH parametric system employs a primary sensor assembly comprised of a glass electrode, a reference electrode, a grounding rod, and a temperature compensator. The glass electrode is permeable to the hydrogen ion and at 25°C produces a potential of approximately 59 millivolts per pH unit.

The reference electrode employed in pH is identical to those employed in ORP and dissolved chlorides. It consists of a silver-silver chloride stem immersed in a solution of KCl saturated with silver chloride maintaining a half cell potential of approximately 220 millivolts at 25°C. The Monel grounding rod serves as a reference to both the glass electrode and the reference electrode. Temperature compensation is effected for cell potential by the application of temperature-sensitive thermistors potted in the Monel rod, so that pH is directly proportional to the concentration of the hydrogen ion.

The glass electrode is extremely fragile. When it is cleaned, great care should be exercised so that the glass tip does not become separated from the glass stem. The reference electrode may have an accumulation of salt at the bottom and when this precipitation is approximately three-fourths of the stem, it should be dissolved by adding distilled water, gently shaking the reference electrode, and discarding.

The electrolyte should be removed with great care to prevent any spillage. Spillage of this highly conductive liquid on the terminal board, for example, will interfere with high-impedance measurements.

The electrolyte is a 4M KCl solution saturated with silver chloride and sold under Beckman label #4787. No other electrolyte should be used in the reference electrodes.

Before pursuing any work, the following instructions should be read carefully. It is expected, however, that after several maintenance visits, these procedures can be streamlined to facilitate field work.

(a) Preliminary Check

1) Observe panel meter deflection and record.

2) Check the electrical calibration at the pH 7 and pH 12 points by inserting the grounding coax cable at the rear of the analyzer deck and employing the push button toggle switch. With push button in and toggle switch down, the panel meter should indicate a pH of 7.

3) Mark with a pencil the orientation of the screw slot on each of the following potentiometers: Z offset, Z-1 scale, R-4, and R-5. Replace the ground coax lead with the signal lead to the pH electrometer and close the drawer. Insert the pH sensor assembly into a beaker of distilled water after cleaning off slime or any other contaminant which has accumulated since the last service call.

The above points should be performed before any further work is attempted with pH parametric systems.

(b) Maintenance and Calibration

Fisher Scientific Buffers are employed as standards for calibration of this instrument.

1) In the measurement of all pH standards, allow approximately a 5-minute response time for each measurement.

2) Clean the pH sensor assembly thoroughly by squirting with distilled water and wiping the glass electrode and reference electrode with a dampened lint-free paper tissue. Remove any accumulation on the underside of the rubber stopper, on the grounding rod, and on the temperature compensator housing. After the pH sensor assembly has been thoroughly cleaned and before other work is performed (i.e., exchange of reference electrode electrolyte), return sensor assembly to its position in the flow cell and observe the panel meter deflection and record.

3) Remove pH sensor assembly from the flow cell, clean and dry, and insert into a beaker of distilled water. Clean flow cell thoroughly with a brush located in the flow cell drawer.

4) If the reference electrode has a sufficient supply of electrolyte, i.e., within an inch and a half below the filling hole at the top, perform the following, employing standards of pH 4, 7, and 10. If the reference electrode requires maintenance, i.e., removal of excess salt precipitation, or addition of Beckman electrolyte, it should be done at this point.

Since these standards are heavily buffered, it is unnecessary to rinse repeatedly. It is required, however, that the sensor assembly be wiped dry before immersing in a standard. Under no circumstance should a dirty sensor assembly be inserted into the standards.

5) Insert the pH sensor assembly into a pH 7 standard, allowing 5 minutes for transients to subside and record the panel meter deflection.

6) If the pH of the river sample has been greater than 7, thoroughly wipe the sensor assembly and insert into the pH 10 standard. Permit a 5-minute response time and record the panel meter deflection. (If the river sample has been consistently less than a pH 7, use a pH 4 standard instead of the pH 10 standard.

7) If the panel meter agrees with both the pH 7 and pH 10 standards, the instrument is calibrated.

8) If the pH indication, with the sensor inserted into the pH 7 standard, shows a difference of more than 0.1 pH units, it will be necessary to adjust the instrument. If the pH reading, with the sensor inserted into the pH 10 standard, shows a difference of more than 0.1 pH units, it will be necessary to adjust the instrument.

9) If the indication for a pH 7 is not in agreement, return the sensor assembly to the pH 7 standard after thoroughly wiping the sensor assembly dry. After transients have subsided, adjust the 7 offset on the front panel to obtain agreement between the buffer and the panel meter.

10) Thoroughly wipe sensor assembly and insert into a pH 10 standard. After transients have subsided and if the panel meter indicates a pH 10, the system is calibrated.

11) If agreement does not exist, adjust the full-scale potentiometer so that the panel meter indicates a pH 10.

12) If the full scale is adjusted, repeat steps 9 through eleven until there is no need for adjustment in the last standard measured.

13) Once agreement exists between the panel meter at a pH of 7 and at a pH of 10, measure a pH of 4 and record its value.

14) All standards have a temperature coefficient so that the temperature must be measured and the pH value adjusted according to the label provided by Fisher Scientific. It is this adjusted value which is employed for calibration. In the above, the nominal values were used for simplicity.

15) Once the system has been calibrated, return the sensor assembly to its position in the flow cell and allow a five-minute response and record panel meter deflection.

16) Employing the grounding coax cable at the rear deck, use the push button and toggle switch to set potentiometers R-4 and R-5 accordingly: with the push button in and the toggle switch down, adjust R-4 so that the panel meter deflects to a pH of 7.

17) With the push button in and the toggle switch up, adjust R-5 so that the panel meter deflects to a pH of 12.

18) With the push button compressed and toggle switch up, adjust R-3a so that the recorder deflects to full scale.

19) Remove grounding cable and return the signal coax cable to the connector at the rear of the deck of the analyzer.

20) R-6 and R-7 are temperature-compensating potentiometers and have been adjusted in the laboratory.

21) Potentiometer R-3b has been adjusted to provide 1000 millivolts to the transmitter.

22) The glass electrode should have a sealant between the bakelite and the glass. The Schneider Instrument Company has employed fingernail polish which has been most effective.

2. Conductivity Parametric System - Introduction

The conductivity parametric system employs a primary sensor assembly comprised of a cell and a temperature compensator. The cell is contained in an epoxy housing and consits of three stacked rings on the inner surface of the longitudinal hole. The center ring is above ground and the top and lower rings are electrically connected and operated at ground potential. The three nickel rings are plated with a platinum black surface.

The conductivity of a water sample is temperature dependent so that thermistors, potted in a Monel rod, are employed to provide a conductivity indication referenced to 25°C. The temperature compensation characteristic of the thermistor network has been adjusted to that of potassium chloride and provides a conductivity correction of approximately 1.84% per degree Centigrade.

The longitudinal hole in the conductivity cell is susceptible to mud and clay formation as well as algae formation and must be thoroughly cleaned before insertion in the primary solutions. However, under no circumstance should the device be cleaned chemically, under power, or with any mechanical abrasive device.

The conductivity parametric system employs an active analyzer having a 1000 cycle oscillator circuit card which produces the reference potential. This potential is coupled through the differential amplifier to ground through the conductivity cell. Temperature compensation adjusts the magnitude of this reference potential and is located in the input circuit.

Before pursuing work with the conductivity parametric system, the following instructions should be read carefully. It should be noted that the conductivity calibration solutions should be properly labeled and always sealed when not in use. When calibrating, place standards conveniently near the sensor assembly with an abundance of tissue for drying the sensor assembly.

In the calibration of the conductivity parametric system, two portions of the same standard are employed. One is labeled a wash solution; the other, the primary standard. When a clean sensor is immersed into a wash, then into the primary standard of the same value, the droplets of solution remaining on the sensor are of approximate equal value and therefore do not contaminate the primary standard.

(a) Preliminary Check

1) Observe panel meter deflection and record.

2) Check the electrical calibration near zero and at full scale by pressing the push button. With the toggle

switch down, the panel meter on the conductivity analyzer will deflect to 1/6 of full scale or 200 μmhos for a 1200 range. With the push button compressed and toggle switch up, the panel meter will deflect to full scale on both ranges.

3) Mark the orientation of the screw slot on the 1/6, full scale, R-4 and R-5 potentiometers.

4) Insert the conductivity probe assembly into a beaker of distilled water and allow to set for several minutes to facilitate cleaning.

These above steps must be performed before any further work is attempted with the conductivity parametric system.

(b) Maintenance and Calibration

1) In the measurement of all primary standards, allow approximately 5-minute response time for each measurement.

2) Thoroughly clean the conductivity probe by squirting water into the hole in the cylinder, being careful not to wet the terminals in the flow cell. Continue to squirt water through this opening until all clay and algae residue have been reasonably well removed. Measure river sample again, permitting a 5-minute response time and observe panel meter deflection.

3) If a conductive clay adheres to the inner surface of the cell, there will be a tendency to record low conductivity at a higher value since the equivalent resistance path between the center ring and the outer two rings has been reduced. The flushing by a squirt bottle will generally wash out this clay. It is necessary to remove the contaminant before the standard solutions are employed.

4) For a full-scale range of 1200 μmhos, conductivity standards of 100, 400, 600 and 1000 can be employed to check the overall calibration. However, only the 100 and 1000 solutions should be employed with 1/6 and FS adjustments. The 1/6 control provides an offset current added to the amplifier output current at the panel meter. It can be employed with either a 100 or 200 μmho standard. These instructions are based upon a 100 μmho standard.

For a full-scale range of 2400 μmhos, standards of 200, 1200, and 2400 μmhos can be used to check the overall calibration. The following steps are applicable to the

0-1200 μmho range. The panel meter deflection for the wash and primary standard solutions should be recorded. Discard wash solutions that deviate by more than 10% from the standard solution.

5) Insert the probe into the 100 wash then into the 100 standard and observe the panel meter deflection. Do not make any adjustment.

6) Thoroughly wipe the probe and blow into the side hole to remove any droplets of standard.

7) Insert probe into a 400 wash, then into a 400 standard and record the panel meter deflection.

8) Wipe probe dry and immerse into a 600 μmho wash then into a 600 μmho standard and record the panel meter deflection.

9) Wipe probe dry and insert into a 1000 μmho wash and then into a 1000 μmho standard and record the panel meter deflection.

10) If the deviation of the panel meter from the standards is within ±12 μmhos, the conductivity parametric system is calibrated. This initial check on all standards prevents the possibility of calibrating with badly contaminated standards or an unclean conductivity cell. (If the panel meter deflection is plotted against the standard solutions, the resulting graph should be a straight line).

11) If the deviation is greater than 12 μmhos, e.g., 50 μmhos, then again insert the conductivity cell into distilled water and try to clean it more thoroughly.

12) Wash probe in a 100 μmho solution then measure the 100 μmho standard and adjust the 1/6 potentiometer to obtain panel meter reading of 100.

13) Remove the conductivity probe, dry with tissue, insert into a 600 μmho wash, remove and dry thoroughly.

14) Insert into a 1000 μmho wash and then into a 1000 μmho standard and observe panel meter deflection.

15) If agreement occurs, the conductivity parametric system is calibrated.

16) If agreement does not exist, adjust the full-scale potentiometer so that the panel meter indicates 1000.

17) Return to the 100 µmho wash through the 400 µmho wash solution after the probe has been thoroughly wiped dry.

18) By inserting into the 400 µmho wash, drying, then inserting into the 100 µmho wash solution, the possibility of contaminating the low calibration standard is reduced.

19) Measure the 100 µmho standard and observe the panel meter deflection. If within ±12 µmhos, the conductivity parametric system is calibrated.

20) If a greater deviation occurs, trim the 1/6 potentiometer to obtain agreement and return to the 1000 µmho standard after drying probe, dipping temporarily into the 600 µmho wash, the 1000 µmho wash and then into the 1000 µmho standard. Dry probe after each dipping into the 600 µmho wash.

21) If the panel meter deflection agrees to each measured standard, the system is calibrated; if not, repeat as above.

22) Repeat until each of the 100 µmho and 1000 µmho standards are measured to within ±12 µmhos. With care and patience, it is possible to obtain a calibration within one-half of one per cent.

23) To set the near zero electrical calibration point, compress the push button and with the toggle switch down, adjust R-4 to obtain 1/12 of full scale.

24) To set the full scale electrical calibration point, compress the push button and with the toggle switch up, adjust R-5 for full-scale deflection.

25) With push button in and toggle switch up, adjust R-3a so that the recorder indicator deflects to full scale for this particular measurement.

26) R-6 and R-7 are temperature compensators and have been adjusted in the laboratory.

27) R-3b provides 1000 mv to the transmitter and has been set in the laboratory.

28) If, with the above calibration procedure it is impossible to effect a calibration, contact the Instrumentation Activity of the Division of Pollution Surveillance. It is anticipated that it will be necessary to replace the conductivity cell once a year.

3. Dissolved Chlorides Parametric System - Introduction

The dissolved chlorides parametric system employs a primary sensor assembly comprised of a silver-silver chloride billet electrode, a silver-silver chloride reference electrode, and a silicon resistor (with positive temperature coefficient) potted in a Monel rod for temperature compensation. A grounding rod located in the sensor assembly acts as ground reference for both the silver-silver chloride billet electrode and the reference electrode.

The sensor assembly is affected by vigorous stirring and by the different contaminants in the sample under test. The analyzer of the dissolved chlorides parametric system is temperature-sensitive so that 15 minutes after calibration adjustment the instrument should be checked in a 2400 μmho standard solution, and lastly, the full scale electrical calibration point should also be checked.

Before any work is attempted, the following instructions should be read carefully. For those who have worked with the systems, a slight deviation from these instructions is expected and encouraged in order to streamline the field work. Note that the chlorides calibration solutions should be properly labeled and placed conveniently near the electrode assembly along with an abundance of tissue for drying the assembly before and after removal from the standards.

(a) Preliminary Check

1) Observe panel meter deflection and record.

2) Check the electrical calibration by grounding the electrometer input with the coax cable on the deck of the analyzer. Do not leave the drawer open long enough to permit it to cool off.

3) With the push button compressed and the toggle switch down, check the low range calibration point at 20 mg/l. Check the high range at 2400 mg/l with the push button in and the toggle switch up. Record the panel meter deflection for both combinations.

4) Mark the orientation of the screw slot on the potentiometers 1/12 and full scale with a pencil for both ranges (i.e., front panel is lower range, deck is high range) and for potentiometers R-4 and R-5.

5) Remove the coax shorting cable and insert the signal cable and quickly close the drawer.

6) Allow 5 minutes before making any measurements to permit temperature recovery.

These points must be performed before any further work is attempted.

(b) Maintenance and Calibration

1) In the measurement of all primary standards, allow a 5-minute response time for each measurement.

2) Thoroughly clean the sensor assembly with distilled water and tissue. In cleaning the chloride sensor assembly, make a record of the type of contamination on the Monel rods and the entire sensor assembly structure. This should include the degree of slime buildup and its characteristics. Such information may be useful to the laboratory personnel. It is suggested that some scrapings of this contaminant be taken to the laboratory periodically for examination.

3) Test chloride electrode for separation from the bakelite housing. A poor sealant has sometimes caused trouble with this electrode. Under such circumstances, water intrusion will cause a potential to be developed in parallel with the half-cell potential of the silver-silver chloride electrode causing an internal loop and an erroneous reading. More effective seals are anticipated on these electrodes in the future.

4) Measure, in order, the 20 mg/l wash, the 20 mg/l standard, the 200 wash, the 200 standard, the 2400 wash, and lastly, the 2400 standard, recording the value of each measurement indicated by the panel meter.

5) If the panel meter deflection for the 200 is less than 100 mg/l, it will be necessary to recondition the surface of the silver-silver chloride electrode even though the electrode may not have a dark gray appearance. (As a matter of practice, it is preferable not to recondition the electrode surface unnecessarily).

6) Wipe entire sensor assembly with tissue.

7) Inspect silver-silver chloride billet electrode and if dark gray in color and if the indication for the 200 mg/l standard solution is less than 100, perform steps 8 through 11.

8) Scrape sides with edge of pocket knife. Emery paper is not desirable as it may cause contaminant to be pushed into the soft surface of the electrode.

9) Insert sensor in standard and observe calibration at low and high chlorides.

10) Place in system and observe new indication.

11) Leave in system for 24 hours before attempting to calibrate. This permits the surface to 'condition' during which time the output will decay exponentially from an excessively high value.

12) If the silver-silver chloride billet electrode is silvery rather than dark, merely wipe the entire sensor assembly with tissue.

13) Remove mercury relay and lay on top of its socket on analyzer chassis. This prevents the automatic range change from occurring when the sensor assembly is removed from the standard solution. Close the analyzer drawer as quickly as possible.

14) Insert the sensor assembly into the 20 mg/l wash solution, carefully stirring manually.

15) Insert into the 20 mg/l standard and let stand for 5 minutes and record indication.

16) If the panel meter deflection does not agree with the standard, adjust the 1/12 control to obtain agreement.

17) Insert sensor assembly into the 200 wash solution.

18) Insert sensor assembly into the 200 standard and observe panel meter deflection and record.

19) If panel meter does not agree, adjust full scale to obtain agreement.

20) If no adjustment is required, the low range is calibrated.

21) If an adjustment is made, dry sensor assembly and insert into the 20 wash solution.

22) Insert into the 20 standard and observe panel meter deflection.

23) If no adjustment at the low end is required, the low range is calibrated.

24) If adjustment is necessary, repeat steps 16 through 21 until the last measurement does not require adjustment.

25) To calibrate the high range, insert the shorting plug into the socket for the mercury relay. Close drawer but do not lock as all high range adjustments are on the deck of the analyzer.

26) Insert the primary sensor assembly into the 200 mg/l wash solution, then into the 200 mg/l standard, and observe panel meter deflection and record.

27) To obtain agreement between the 200 mg/l and the panel meter, adjust the 1/12 potentiometer located adjacent to the yellow pin jack on the deck of the analyzer.

28) Insert the sensor assembly into the 2400 mg/l wash solution, then into the 2400 mg/l standard and obtain agreement between the panel meter deflection and the standard by adjusting the FS potentiometer adjacent to the 1/12 adjust on the deck of the analyzer.

29) If no adjustment is required for the 2400 mg/l measurement, the high range is calibrated.

30) If an adjustment on the 2400 mg/l range is required, wipe sensor assembly dry, rinse in the 200 mg/l wash solution, insert into the 200 mg/l standard and check panel meter deflection.

31) If the panel meter reads 200 mg/l, no adjustment is required and the high range is calibrated. If adjustment is necessary, repeat steps 27 through 30.

32) The R-12 potentiometer located on the deck of the analyzer adjusts the pull-in voltage of the mercury relay. Replace the shorting plug with the mercury relay and adjust R-12 so that in the 200 mg/l solution the relay does not pull in; i.e., it should pull in at about 220 mg/l.

33) Potentiometer R-11 adjacent to R-12 determines when the mercury relay R-1 drops out. Adjust R-11 so that relay 1 drops out at about 180 mg/l.

34) Automatic change from the low range to the high range can be observed by lifting the sensor assembly out of the standard causing full-scale deflection. As the panel meter passes 220, the red light goes on at which time change-over occurs and the gain of the amplifier is reduced by an order of magnitude.

35) Insertion of the sensor assembly into a chloride wash solution of 20 mg/l from the high range (i.e., with the sensor assembly in air) causes a down-scale reading to occur. As the panel meter passes the second major division, or about 180 mg/l, the red light should go off and the panel meter will then go back upscale momentarily and then drift back down toward an indication of 20 mg/l.

36) The accuracy of the chlorides parametric system is 5% of full scale, or 12 mg/l on the 240 range and 120 mg/l on the 2400 range. It is possible, however, to obtain a 1% calibration.

37) Potentiometers R-7a and R-7b are in parallel with the temperature compensators and are laboratory adjustments.

38) Resistors R-4 and R-5, located on the analyzer deck to the rear left, are employed to adjust the electrical calibration points at 20 mg/l and at 2400 mg/l. For these adjustments, the electrometer input is grounded when the test coax cable is inserted in the white pin connector. After the calibration with chemical standards has been completed, check the 1/12 calibrate (push button in, toggle switch down) by inserting the grounding coax cable in the signal input at the rear of the analyzer. Do not allow connector to touch fuses on adjacent analyzer.

39) Adjust R-4 so that panel meter reads 20 on the 240 mg/l range.

40) With toggle switch up and push button in, adjust R-5 to obtain a full-scale deflection of 2400 mg/l.

41) Note, both R-4 and R-5 have the capability of producing 0-1000 mv in the output.

42) Resistors R-3a and R-3b located immediately behind the panel meter provide input signals to the recorder and to the transmitter, respectively. Center tap of R-3a provides 50 mv to the recorder for full scale on either range and R-3b provides 1000 mv full scale for either range to the transmitter.

43) When the chemical and electrical calibration has been completed, use the electrical calibrate position (coax ground cable, push button in, toggle switch up) and adjust R-3a so that the recorder agrees with the panel meter deflection.

44) R-3b has been set to provide a 1000 millivolt signal to the transmitter on the high range. Note that both R-3a and R-3b are always in the output string regardless of range.

4. Dissolved Oxygen Parametric System - Introduction

The dissolved oxygen parametric system is comprised of a gold-silver electrode assembly and a temperature compensator and associated active analyzer. The polarographic principle is employed and molecular oxygen, as a dissolved gas, diffuses through a polyethylene membrane and is reduced at the gold cathode.

The reduction of oxygen is effected through the application of -0.8 volts derived from a Zener-regulated voltage supply.

The permeability of the membrane material is temperature-sensitive so that the diffusion of oxygen through it increases in an exponential manner with temperature resulting in an exponential increase in current output of the electrode assembly. Thermistors, having a similar but negative exponential characteristic with temperature, are employed in the feedback of an amplifier so that the current impedance product for a given dissolved oxygen constant is a constant for all temperatures. Hence, the analyzer produces an output proportional to dissolved oxygen in milligrams per liter.

(a) Preliminary Check

1) Observe panel meter deflection and record.

2) Check electrical calibration through the operation of the push button switch and the toggle switch located on the front of the analyzer immediately below the panel meter.

3) Generally, the low indication is set for 4.0 mg/l and the high indication is set for 24 mg/l.

4) Mark the orientation of the screw slot on the full-scale potentiometer adjustment and on the potentiometers R-4 and R-5 used for the electrical calibration check points.

5) Do not remove the dissolved oxygen sensor assembly from its flow cell (see b below). Draw two samples for Winkler determinations from an adjacent flow cell by employing the small plastic hose provided with the system.

6) Record the dissolved oxygen panel meter deflection during the time that the BOD bottle is being filled with the sample.

7) If a sudden excursion in dissolved oxygen occurs, as noted by the panel meter, discard the sample and start over as it will not be a valid comparison.

8) Insert the manganous sulfate and the alkaline iodide into the BOD bottles and permit samples to settle during which time (b) can be performed. An average value of these Winkler determinations will be used and is referred to as the first set of Winkler determinations.

(b) Maintenance and Calibration

1) Gently remove the dissolved oxygen electrode assembly and observe its characteristics; i.e., slime buildup, precipitation internal to the electrode, cap of electrode, and cables to sensor assembly.

2) Gently wipe slime from the membrane directly opposite the gold cathode and gently reinsert the sensor assembly into the flow cell.

3) Note panel meter deflection for this partial cleaning and compare with panel meter deflection recorded in (a)6 above.

4) Remove sensor assembly and thoroughly clean membrane by squirting with distilled water and pulling slimy material from the membrane where it has adhered. Note that the membrane characteristics permit clinging of the different algae formations and a rather vigorous cleaning with a squirt bottle may be necessary. Do not clean with an abrasive as it may puncture the membrane.

5) Clean entire stem of the dissolved oxygen electrode, the temperature compensator, and the temperature probe which is integral with the dissolved oxygen electrode assembly.

6) Clean dissolved oxygen flow cell.

7) Return dissolved oxygen electrode assembly to its flow cell position and allow 5 minutes for transients to subside. (Note that when the dissolved oxygen electrode is exposed to the atmosphere there is a tendency to an increase in dissolved oxygen indication when the river sample is below 8 ppm).

8) Draw a third and a fourth sample for two more Winkler determinations from the same flow cell that the first set had been drawn and insert the manganous sulfate and alkaline iodide chemicals and permit to settle. The average value of these two determinations will be employed and is referred to as the second set of Winkler determinations.

9) While the sample is being drawn, record the panel meter deflection.

10) Complete the first set of Winkler determinations by adding sulfuric acid and titrating, making certain of the normality of the titrant. Obtain an average of the two Winkler determinations.

11) Record this value of dissolved oxygen and compare with the panel meter deflection recorded in (a)6. This is the comparison data before maintenance.

12) Complete the second set of Winkler determinations by adding the acid and titrating and compare the average value with the data recorded in 9.

13) If the comparison in 12 is within a percent of full-scale value, the system is calibrated.

14) If, in the first comparison the panel meter deflection is lower than the Winkler and in the second comparison the panel meter agrees to the Winkler, no calibration is necessary and the deviation in panel meter is merely the error caused by slime buildup on the electrode. This will normally be the case for the first four to six weeks of operation.

15) If, in the first case the Winkler and panel meter are in agreement but in the second case the panel meter exceeds the Winkler, there is a possibility that the electrode has become defective. If this increase is less than 4%, merely reduce the full-scale adjustment to obtain agreement between the panel meter and the Winkler.

16) If, in both cases the panel meter is less than the Winkler and in the second case if the error is less than 5%, adjust the full scale to obtain a panel meter deflection that coincides with the average of the second set of Winkler determinations.

17) After six weeks a silver oxide and silver chloride precipitation may form near the cathode causing a loss in sensitivity of the sensor. A buildup of silver chloride and silver oxide on the anode also causes a loss in sensitivity after a period of time. It is also possible that a peripheral leakage may occur which causes an excessively high dissolved oxygen indication. When these conditions exist, replace the dissolved oxygen electrode and arrange for the return of the used electrode to the Instrumentation Activity, Division of Pollution Surveillance, 1014 Broadway, Cincinnati, Ohio.

18) After a calibration has been effected, again run another Winkler and record the panel meter deflection, observing whether agreement exists.

19) Finally, adjust R-4 with the push button in and the toggle switch down to obtain an electrical calibration at 4 mg/l.

20) With the push button in and toggle switch in the up position, adjust R-5 to 24 mg/l.

21) With the full-scale calibration condition, observe the recorder deflection and adjust R-3a to obtain full-scale deflection.

22) Potentiometers R-6 and R-7 are employed for temperature compensation and have been set in the laboratory.

23) Potentiometer R-3b provides 1000 mv to the transmitter and has been set in the laboratory.

24) It should be noted that the wires to the sensor assembly are color-coded. Also, each terminal is numbered and each wire has a corresponding identification. The gold cathode has a yellow wire attached to it and the anode, which is comprised of four solid silver rods connected externally by a Monel ring, is electrically cabled to the summing point through a white-black cable.

25) In some instances, chemical interference with the Winkler procedure may prevent a valid comparison with the monitor. In this instance, the monitor can be checked by measuring the dissolved oxygen concentration in a sample of aerated, distilled water as noted below.

26) Gently stir sample with sensor assembly until steady-state conditions have been reached. Allow approximately 5 minutes, but do not expose the dissolved oxygen electrode to the atmosphere unduly as this will increase the transient response.

27) Observe panel meter deflection and record.

28) Obtain two samples for Winkler determination of dissolved oxygen as noted previously. (See Appendix).

29) Compare panel meter deflection with Winkler determination. Adjust the full scale potentiometer so that the panel meter agrees to the Winkler determination.

30) Adjust the electrical calibration according to steps 19 and 20.

5. Oxidation-Reduction Potential Parametric System - Introduction (Experimental)

The ORP parametric system measures the potential of the water sample under test with a platinum inlay electrode and a silver-silver chloride electrode. Both electrodes are referenced to a Monel rod at ground potential. The reference electrode is identical to those used in the pH and chlorides measurements.

Since the condition of the water affects the polarity and, to some extent, the magnitude of its potential, the ORP parametric system is provided with two ranges: -600 through 0 to +600 millivolts, and, 0 to +1200 millivolts. A center zero is obtained by adding a 600 millivolt offset potential with the zero control on the front of the analyzer.

However, before changing ranges, those responsible for data processing should be contacted so that a -600 millivolt offset can be added in the subroutine.

Similar to other sensors, the ORP sensor is affected by pollution parameters and natural river sample constituents. However, the output of this experimental parameter may either increase or decrease, dependent upon the contaminant. Hence, periodic cleaning of the sensor assembly with tissue is necessary.

Before attempting calibration, the following instructions should be read carefully.

a) Preliminary Check

1) Observe panel meter deflection and record.

2) Clean the platinum electrode and entire sensor assembly and observe the panel meter deflection and record. The difference between 1 and 2, assuming constant sample potential, was caused by the contaminant on the sensor.

3) Check the electrical calibration points by using the coax grounding cable, push button, and toggle switch. With the push button in and the toggle switch down, the panel meter should indicate zero. With the push button in and the toggle switch up, the panel meter should indicate full scale.

The above points should always be performed before performing the following work.

(b) Maintenance and Calibration

1) With the coax grounding cable connected to the white pin jack at the rear on the deck of the analyzer and with the push button compressed and toggle switch down, adjust R-4 for zero output potential of the P-2 amplifier. This potential is measured between the green and white pin jacks.

2) Under the same conditions as 1 above, adjust the zero control on the front panel of the analyzer for zero panel meter deflection.

3) Under similar conditions as 1 above but with the toggle switch up, adjust R-5 so that the output potential of the P-2 is -1200 millivolts.

4) Under the same conditions as 3, adjust the gain potentiometer FS located on the front panel so that the panel meter indicates full scale.

5) With coax grounding cable inserted, push button compressed and toggle switch up, adjust R-3a so that the recorder deflects to full scale.

6) Remove coax grounding cable and insert the signal coaxial cable and observe panel meter deflection with sensors immersed in river sample.

7) Wash and dry sensor assembly and measure a pH 4 standard (500 milliliters) containing 0.5 grams of quinhydrone at 25°C. The panel meter should indicate approximately +263 millivolts.

8) If the panel meter deflection in 7 deviates by more than ±5% of full scale, repeat from (b)1.

6. Solar Radiation Intensity Parametric System - Introduction

An Eppley Pyrheliometer is employed as the sensor in the solar radiation intensity parametric system and is comprised of a sodium glass enclosed thermal pile mounted on a three-legged adjustable stand with leveling screws. The model installed at the Arlington Memorial Bridge has 50 thermocouple junctions. The hot junctions are located in the black ring, and the cold junctions are located in the center magnesium-oxide coated circular disc. The sensor has a 70% transmittance between 0.32 and 4.20 microns and produces about 7.5 millivolts per calorie per square centimeter per minute.

The bulb is sealed and is filled with dry air. However, leakage has occurred in the past and when it does, the output is greatly reduced dependent somewhat upon the climatic conditions.

The output of the pyrheliometer is temperature compensated by a thermistor-resistor network located in a junction box directly beneath the sensor. This box has a rubber gasket seal but should be inspected every couple of months and checked for salt formation.

The sensor leads enter the rear of the flow cell cubicle and can be removed for measuring the millivolt potential at any given time with a potentiometer.

Calibration of the sensor is a laboratory function and can be performed every 12 to 18 months dependent somewhat on the needed accuracy for this particular measurement. The calibration check summarized below does not have to be performed very frequently due to the nature of the measurement. The sensor is very fast so that a cloud passing overhead may cause the panel meter to drop suddenly from 1.20 to 0.30 on a sunny day.

Before performing any adjustment on a solar radiation intensity parametric system, the following instructions should be read carefully.

(a) Preliminary Check

1) Observe panel meter deflection and record. Whether this piece of data will be useful after a calibration is dependent upon the time required for the work and the time of the day.

2) Check the electrical calibration points by employing the push button switch and the toggle switch. With the push button pressed and the toggle switch down, the panel meter should indicate zero. With the push button in and the toggle switch up, the panel meter should indicate full scale.

The above points should be performed before pursuing the following.

(b) Maintenance and Calibration

1) Clean terminals for the sensor input at the flow cell to remove any salt residue or water droplets.

2) Wipe the sensor mounted on the piling with a dry, clean cloth and observe the relative position of the sun, sensor, and any cloud formation. Note the sensitivity on the band about the base of the Eppley Pyrheliometer.

3) Record panel meter deflection.

4) If initial electrical calibration checks are respectively zero and full scale, measure the millivolt output of the solar intensity sensor with a potentiometer and call this potential V_1. V_1 is to be measured with the sensor conductors connected to the terminal board.

5) Disconnect the sensor leads from the terminal board at the flow cell and, using a Rubicon potential source, apply potential V_1 to the input terminals of the solar intensity analyzer and obtain a panel meter deflection.

6) Compare the panel meter deflection in 5 with 3. There should be agreement between these two measurements.

7) Adjust the Rubicon output potential until the panel meter reaches full scale or 2.4 calories per square centimeter per minute.

8) The ratio between the millivolt potential and the panel meter should be equal to the sensitivity of the sensor in millivolts per calorie per square centimeter per minute.

9) If the ratio in 8 is different from the sensitivity, adjust the Rubicon output potential to the product of the pyrheliometer sensitivity and full scale panel meter indication (7.45 x 2.4 = 17.9 millivolts) and apply to the terminals as in item 5.

10) Adjust the full scale calibrate potentiometer to obtain 2.4 on the panel meter.

11) If a calibration cannot be obtained, contact the Instrumentation Activities, Division of Pollution Surveillance, 1014 Broadway, Cincinnati, Ohio.

12) Replace the sensor leads and observe panel meter deflection.

13) Adjust electrical calibrate potentiometers by compressing the push button and with the toggle switch down, adjust R-4 for zero panel meter deflection.

14) With the push button compressed and the toggle switch up, adjust R-5 for full scale and adjust R-3a for recorder full-scale deflection.

15) R-3b has been set for 1000 millivolts full scale at the laboratory.

16) If the electrical calibration points noted by 4 are respectively zero and full scale in a subsequent maintenance call, conclude that the analyzer is in calibration.

7. Temperature Parametric System - Introduction

The temperature parametric system employs a primary sensor assembly comprised of a silicon resistor (with positive temperature coefficient) potted in an epoxy within a Monel rod. Although integral with the assembly for dissolved oxygen, the temperature probe can be inserted into any flow cell, but its physical location might be limited by the length of the gray leads terminating on the terminal board.

Temperature is measured in degrees Fahrenheit as noted by the panel meter which is marked 0-120°F. Selection of panel meter values is a matter of circuit symmetry, as it is not expected to measure temperature over this entire range.

The sensor for temperature is not affected by any of the contaminants, but periodically it should be checked with different samples at different degrees, as noted below.

(a) Preliminary Check

1) Observe panel meter deflection and record.

2) Check calibration check points employing the push button and toggle switch located on the front of the analyzer directly below the panel meter.

3) The zero check point occurs with the push button pressed and with the toggle switch in the down position. The full-scale calibration point occurs with the push button pressed and the toggle switch in the up position. The push button removes the silicon resistor from an input bridge circuit and substitutes the two potentiometers R-4 and R-5 selected with the toggle switch.

4) Both the low range calibration point and the full-scale calibration point should be recorded as a matter of practice.

5) Mark the orientation of the screw slot on the zero adjust, full-scale adjust, and potentiometers R-4 and R-5.

6) Measure the temperature of a flow cell with a thermometer. Record this temperature and the panel meter deflection.

These preceding points must be performed before any further work is attempted.

(b) Maintenance and Calibration

1) Measure the sample with a thermometer and record the temperature.

2) Record the panel meter deflection.

3) If the deviation between the thermometer and panel meter is greater than 1% of full scale, or approximately 1.2°F, and if the water temperature is 40°F or more, adjust the full-scale potentiometer to obtain agreement. Note that the accuracy of the thermometer employed must be ±0.25°F or better. If the water temperature is below 40°F, adjust the front panel "0" control.

4) With the push button compressed and the toggle switch down, adjust R-4 for zero panel meter deflection.

5) With the push button compressed and the toggle switch up, adjust R-5 so that a panel meter deflection of 120°F is obtained.

6) With the push button compressed and the toggle switch up, adjust R-3a so that the recorder is at full scale corresponding to full scale panel meter deflection.

7) Periodically (every 3-4 months) check the temperature parametric system for overall accuracy at the following temperatures: 33°F, 60°F and 100°F. These temperatures are obtained with an ice bath, river water, and a hot plate.

8) Measure the cold sample, river sample and hot sample with an accurate (±0.25°F) thermometer and record. If deviations exceeding 1% occur, calibrate the instrument as follows:

9) Measure the cold temperature with the thermometer strapped to the temperature probe. Adjust the "0" front panel control to obtain agreement between the panel meter and the thermometer.

10) Measure the river sample and record.

11) Measure the hot sample and adjust the full-scale potentiometer on the front panel so that the panel meter agrees with the thermometer.

12) Measure the river sample and return to the cold sample.

13) If no adjustment is necessary (i.e., the deviation is less than 1.2°F), the instrument is calibrated. If an adjustment is necessary in the cold temperature, repeat from 10 above.

14) The instrument is calibrated when each measurement is within ±1.2°F of the thermometer reading.

15) In checking the temperature parametric system for the cold, medium and hot temperatures, the temperature probe can be employed to stir the water to eliminate temperature gradients within the beaker. The thermometer used to obtain a peripheral measurement can be strapped to the temperature probe or held adjacent to it when making the comparative readings.

16) Due to the thermal capacity of the epoxy within the Monel housing, the temperature response is not instantaneous but requires approximately 2 minutes going from the low temperature to room temperature and then on to the high temperature.

17) Repeat steps 4, 5, and 6.

18) Potentiometer R-3b has been set to provide 1000 millivolts to the transmitter.

F. Appendix I

1. Calibration Accessories, Hardware

(a) Electronic Instruments:

Simpson Multimeter, Model 269.

Rubicon potentiometer and millivolt potential run-up box (Honeywell Instrument, Model 2720).

(b) Small Tools:

Wrench, Allen
Wrench, Crescent, 8"
Long-nose pliers, Boker 730-7
Short-nose pliers, Boker ST703-6
Curved-nose pliers, Boker 708-6
Side cutters, Boker 71-4 1/2
Side cutters, Boker 91-6
Screwdriver, Xcelite R188
Screwdriver, Xcelite R5325
Solder, resin core
Soldering iron, 25 Watt

2. Calibration Standards for Parametric Systems

(a) pH:

Buffer solution (Fisher Scientific): pH 4, pH 7, pH 10; 1 qt. each.

Jars, wide mouth, 16 oz.; 3 each.

(b) Conductivity:

Standard solutions: 100, 400, 600, 1000, 1200 and 2400 micromhos, depending upon range; 2 qts. each.

Jars, wide mouth, 32 oz.; 2 for each standard solution.

(c) Dissolved Chlorides:

Standard solutions: 20 mg/l, 200 mg/l and 2400 mg/l; 2 qts. each.

Jars, wide mouth, 32 oz.; 2 for each standard solution.

(d) Dissolved Oxygen: (For preparation of reagents, see Standard Methods, 12th Edition, p 406):

Manganous sulfate solution in 250 ml plastic bottle.
Standard sodium thiosulfate in 500 ml plastic bottle (or 1 qt. of PAO).
Concentrated sulfuric acid in 250 ml glass bottle.
Potassium iodide solution in 250 ml plastic bottle.
Thyodene powder in 50 ml vial.
Bi-iodate solution in 50 ml plastic bottle.
Pipettes, automatic, 2 ml; 3 each.
Flask, 500 ml Erlenmeyer.
Flask, 200 ml volumetric.
BOD bottles; 3 each.
Pipette, 20 ml.
Burette, 50 ml, and support stand.

(e) Oxidation-Reduction Potential:

Quinhydrone powder; 4 oz. jar.
Buffer solution (Fisher Scientific), pH 4; 1 qt.

(f) Temperature:

Thermometer, 30°F to 130°F, 0.2°F intervals.

G. Appendix II: Winkler Procedure for Determining Dissolved Oxygen (Azide Modification) from Standard Methods, 12th Edition, p. 408:

1. Collect a 300 ml sample in a BOD bottle from a flow cell. This sample can be obtained from the stainless steel valve with an attached 6-inch hose mounted on the front panel of the flow cell drawer or the sample can be obtained by using a 12-inch long small diameter rubber hose and siphoning a sample from the flow cell adjacent to the dissolved oxygen flow cell. Place the BOD bottle in a 3000 ml beaker and insert the hose so that it rests on the bottom of the bottle. Allow three sample exchanges in the BOD bottle and make certain that no air bubbles are trapped in the sample and that the sample does not become aerated.

2. Add 2 ml each of manganous sulfate solution and alkaline iodide solution. These reagents should be added below the surface of the sample and the bottle rapidly recapped. Rinse overflow of sample from the neck and use a tissue to absorb any droplets of sample.

3. Invert the sample several times and after the precipitate has settled half way to the bottom, again gently invert the bottle several times and allow time for a second settling.

4. When the precipitate has settled to about half of the depth of the BOD bottle, add 2 ml of concentrated sulfuric acid. Replace the stopper and rinse the excess sample accumulated on top of the stopper and invert several times until the precipitate is completely dissolved in the BOD bottle. Hold with a tissue to absorb droplets of sample.

5. Insert the rubber-collared volumetric flask into the BOD bottle; invert, and fill completely to the 203 ml cutoff mark.

6. The remaining sample in the BOD bottle can be used to rinse the Erlenmeyer flask.

7. Transfer the 203 ml of the treated sample into the Erlenmeyer flask and titrate with standard 0.025 normal sodium thiosulfate to a pale yellow color. Add a scoop of thyodene powder and continue the titration, slowly swirling the Erlenmeyer flask. Continue the titration until the first disappearance of the blue color occurs. The volume of standard sodium thiosulfate required is the dissolved oxygen concentration of the sample. (In this procedure, Phenylarsene Oxide (PAO) can be employed instead of the thiosulfate).

8. If the normality of the thiosulfate is less than 0.025, multiply the volume used by the ratio of its normality to 0.025.

9. To establish the accuracy of the titration, add drops of bi-iodate solution to the sample after the end point has been reached. Each drop of bi-iodate solution is approximately 0.035 ml. When the blue color again appears throughout the sample, subtract the volume of bi-iodate added from the original volume of thiosulfate; the difference is the true concentration of dissolved oxygen in the sample.

10. Rinse all glassware with distilled water and store in an inverted position. The glassware can be stored on a board containing cylindrical pegs similar to standard laboratory drying boards.

11. Cap all reagents and store automatic pipettes in independent rinse (distilled) water.

H. Appendix III: Spare Parts List (first level):

1. Sensors, 1 each, unless otherwise noted:

pH Glass Electrode, #19500 Beckman Instruments.
Reference Electrode, AgAgCl, #19755 Beckman Instruments.
Silver-silver Chloride Billet Electrode, Beckman Instruments.
Dissolved oxygen Electrode, Schneider Instrument Company.